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Adhesive Properties of Ice, Part II

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PREFACE

This is one of a series of reports of work performed on USA SIPRE Project 022.1.004, Thickness and strength of ice surface layers. The purpose of these investigations is to elucidate the mechanism responsible for the adhesive properties of ice.

Work on this project was performed by Dr. H. H. G. Jellinek, physical chemist. Mr. G. M. Walker, research assistant, performed the shear and tensile experiments. Mr. W. Banks and Mr. R. Klaub, Technical Equipment Branch, assisted in constructing the apparatus. Work on this project was performed for USA SIPRE's Basic Research Branch, Mr. J. A. Bender, chief.

This report has been reviewed and approved for publication by the Office of the Chief of Engineers.

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Colonel, Corps of Engineers
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CONTENTS

	Page
Preface	ii
Summary	iv
Introduction	1
Experimental	1
Materials	1
Apparatus	1
Experimental results	2
Ice/metal interface	2
Ice/quartz interface	5
Discussion	8
References	10

ILLUSTRATIONS

Figures

1.	Modified shear apparatus	2
1a.	Details of stainless steel cylinder	2
2.	Typical stress vs time curves for stainless steel disks	4
3.	Relationship between average adhesive strength and rate of shear for snow-ice/stainless steel	5
4.	Adhesive strength vs time for snow-ice/fused quartz optical flat	6
5.	Relationship between average adhesive strength and rate of shear for snow-ice/fused quartz optical flat	7
6.	Typical recordings of force as a function of time for snow-ice/fused quartz optical flat	7

Tables

I.	Adhesive strength of snow-ice sandwiched between stainless steel plates	2
II.	Tensile strength experiments at -4.5C	3
III.	Adhesive strength of snow-ice sandwiched between stainless steel disks of mirror polish	5
IV.	Adhesive strength	6
V.	Tensile strength experiments on the system snow-ice/fused quartz	8

SUMMARY

Adhesive strength of ice for the systems ice/stainless steel and ice/optically flat fused quartz has been investigated as a function of surface roughness of steel surfaces and rates of shear for steel and quartz. The adhesive strength decreases with decreasing roughness of steel surfaces and the force vs time curves for smooth steel plates resemble those of two solids sliding over each other with a liquid layer sandwiched between them. This is particularly so in the case of quartz. The adhesive strength as a function of rate of shear is linear both for ice/stainless steel and ice/quartz; however, there are indications of yield values. The experimental results are in agreement with the assumption of a liquidlike layer on ice. Ratios of viscosity coefficient to the thickness of the layer have been evaluated for both systems and viscosity coefficients are estimated. The importance of interfacial free energy considerations is pointed out.

ADHESIVE PROPERTIES OF ICE, PART II *

by

H. H. G. Jellinek

INTRODUCTION

A previous report (Jellinek, 1957a) dealt with shear experiments on ice sandwiched between stainless steel or polymer disks. The adhesive strength was shown to increase rapidly and linearly with decreasing temperature. In the case of ice/stainless steel, pure adhesive breaks were observed down to a temperature of about -13C, where the adhesive breaks changed abruptly to cohesive breaks, which were only slightly dependent on temperature. The system ice/polystyrene also showed a strong linear temperature dependence of the adhesive strength.

In contrast to shear experiments, tensile experiments (Jellinek, 1957b) showed that the adhesive strength under tension is at least 15 times larger than the adhesive strength obtained from shear experiments. This large discrepancy was explained by the assumption of a liquidlike layer between ice and the solid interface. In case of tension, this liquidlike layer is held together by surface tension forces, whereas in shear only frictional forces, which are of much smaller magnitude, have to be overcome.

The present report deals with shear experiments on the system ice/stainless steel and ice/optically flat fused quartz as a function of the rate of shear and also as a function of the roughness of the steel surface. The experiments reported here substantiate the assumption of a liquidlike layer at an ice/solid interface.

EXPERIMENTAL

Materials

Stainless steel was grade 304/A, the same material as used in previous experiments (Jellinek, 1957b). Snow-ice was prepared as described previously (Jellinek, 1957b). Optically flat fused quartz disks (flat to 1/5 waveband) were obtained from the Crane Packing Company. Cleaning fluids such as methanol and benzene were of reagent grade.

Apparatus

The tensile apparatus was the same as used previously, (Jellinek, 1957b). The shear apparatus (Jellinek, 1957a) was modified (Fig. 1, 1a). A quartz optical flat (1) of 1 in. diam is mounted in a stainless steel cylinder, which is lined with Teflon to allow for temperature contraction of the steel. The steel cylinder has a hook attached to it for fastening a chain transmitting the force to a Baldwin load cell (5). The force acts in line with the surface of the quartz flat. Ice (2) of 1 to 2 mm height is sandwiched between the quartz flat with a somewhat larger diameter than the ice layer and a stainless steel disk with the same diameter as the ice layer. The quartz flat and the lower steel cylinder are located on a large steel cylinder (6 and Fig. 1a). The surface of this steel cylinder is lined with Teflon (see Fig. 1a) and the underside of the small steel cylinder in Figure 1 is also lined with Teflon to reduce friction to a minimum.

A Statham strain gage (4), Transducer Model G 7A-0.15-350, measures the movement of the quartz disk, being in contact with it by means of a small rod. The load cell and strain gage were calibrated using weights and a micrometer screw respectively; proportionality was found between electrical output and load or displacement. The gages were connected to Leeds and Northrup recorders. The procedure of preparing the snow-ice layer and the cleaning procedures are described in a previous report (Jellinek, 1957a). A weight of 500 g plus the steel rod were placed on the steel/ice/quartz sandwich for good bonding, but the weight was taken off before starting the shear experiment.

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* For Part I, see Research Report 38 (1957).

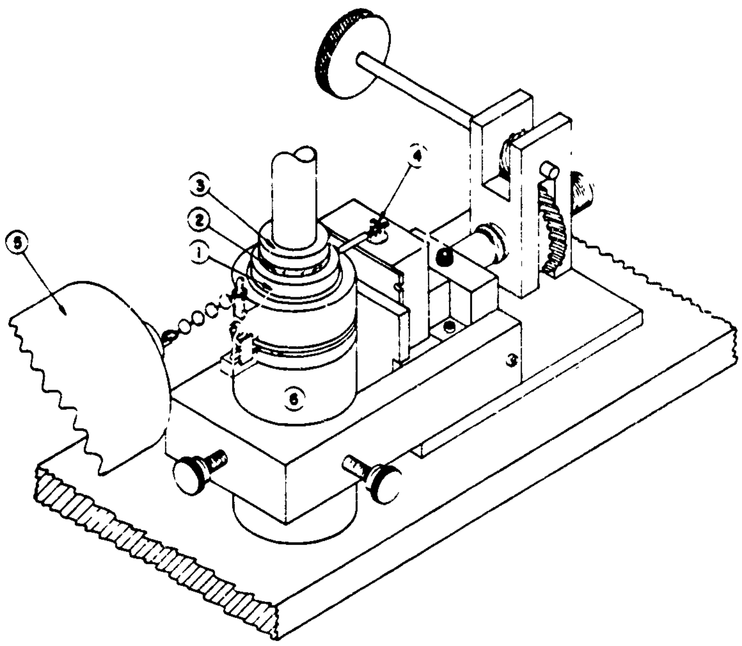

Figure 1. Modified shear apparatus. (1) Quartz optical flat (1 in. diam); (2) Snow-ice layers; (3) Stainless steel disk; (4) Statham strain gage; (5) Baldwin load cell; (6) Stainless steel cylinder.

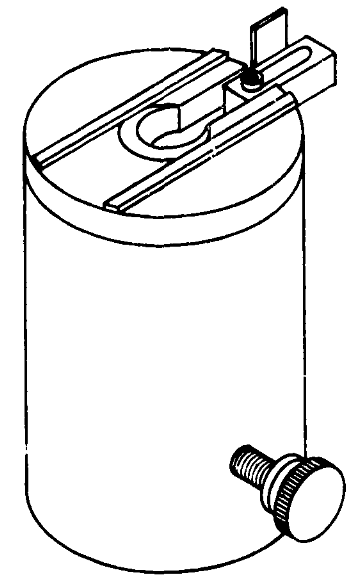

Figure 1a. Details of stainless steel cylinder (No. 6 in Fig. 1).

EXPERIMENTAL RESULTS

Ice/metal interface

Previously (Jellinek, 1957a) all experiments were carried out with one type of polished surface. In the present work, a number of experiments were performed with stainless steel surfaces of different degrees of roughness:

(a) Stainless steel disks turned on the lathe without further polishing.

(b) Finely polished disks (mat surface finish). These surfaces showed a very large number of small pits upon microscopic examination.

(c) Bright mirror polish. These surfaces showed an appreciably smaller number of pits. Profilometer needle readings showed an uneveness of about 5 to 7μ in. The results are given in Table I.

Table I. Adhesive strength of snow-ice (density 0.888 g/cm^3) sandwiched between stainless steel plates, from shear experiments at -4.5C. Cross-sectional area 6.16 cm^2. Height of snow-ice layer 0.1 to 0.2 cm.

(a) Rough plates

Average rate of travel up to maximum strength 5.9 x 10^{-3} cm/sec. Average rate of loading 0.27 kg/cm^2-sec.

Test no.	1	2	3	4	5	6	7	8	9	10	11	12
Adhesive strength (kg/cm^2)	4.1	5.8	2.9	6.4	5.6	8.1	7.2	7.3	6.0	7.3	7.4	5.4

Mean adhesive strength 6.1 kg/cm^2; standard deviation ±1.46 kg/cm^2. Average time of travel 26.5 sec. Average distance travelled 0.16 cm.

Table I. (continued)

(b) Polished plates (mat finish)

Average rate of travel up to maximum strength 5.4 x 10^{-3} cm/sec. Average rate of loading 0.16 kg/cm²-sec.

Test no.	1	2	3	4	5	6	7	8	9	10	11	12
Adhesive strength (kg/cm²)	2.3	2.1	2.5	3.0	3.2	2.5	3.2	2.2	3.1	2.6	2.8	2.9

Mean adhesive strength 2.7 kg/cm²; standard deviation ±0.37 kg/cm². Average time of travel about 16 sec. Total distance travelled 0.086 cm.

(c) Bright mirror polish

Average rate of travel up to maximum strength 5.8 x 10^{-3} cm/sec. Average rate of loading 0.12 kg/cm²-sec.

Test no.	1	2	3	4	5	6	7	8	9	10	11	12
Adhesive strength (kg/cm²)	0.38	0.44	0.75	0.73	0.36	0.36	1.16	0.77	0.65	0.88	0.75	0.89

Mean adhesive strength 0.68 kg/cm²; standard deviation ±0.24 kg/cm². Average time of travel similar to the previous experiments.

The stress vs time curves for the steel disks with bright mirror polish are characteristically different from those for experiments carried out with disks with rough surfaces and mat polish. A few typical curves are shown in Figure 2, which also includes a curve for the quartz optical flat. It is seen that after a rapid increase to a maximum value, the stress decreases comparatively slowly in the case of the mirror polished disks.

A number of tensile experiments were carried out using the mat-polished and the mirror-polished disks to ascertain whether the surface finish has any influence (Table II).

Table II. Tensile strength experiments at -4.5C. Cross-sectional area 3.14 cm². Height of snow ice cylinder 1 cm.

(a) Mat finish

Test no.	1	2	3	4	5
Tensile strength (kg/cm²)	14.3	10.5	16.9	11.9	7.6

Mean tensile strength 12.2 kg/cm²; standard deviation ±3.2 kg/cm².

(b) Mirror finish

Test no.	1	2	3	4	5	6	7
Tensile strength (kg/cm²)	9.6	7.1	11.6	12.4	10.5	8.4	9.6

Mean tensile strength 9.9 kg/cm²; standard deviation ±1.7 kg/cm².

The type of cohesive breaks were similar to those reported previously (Jellinek, 1957b); also the magnitude of the tensile strength values is close to those reported previously.

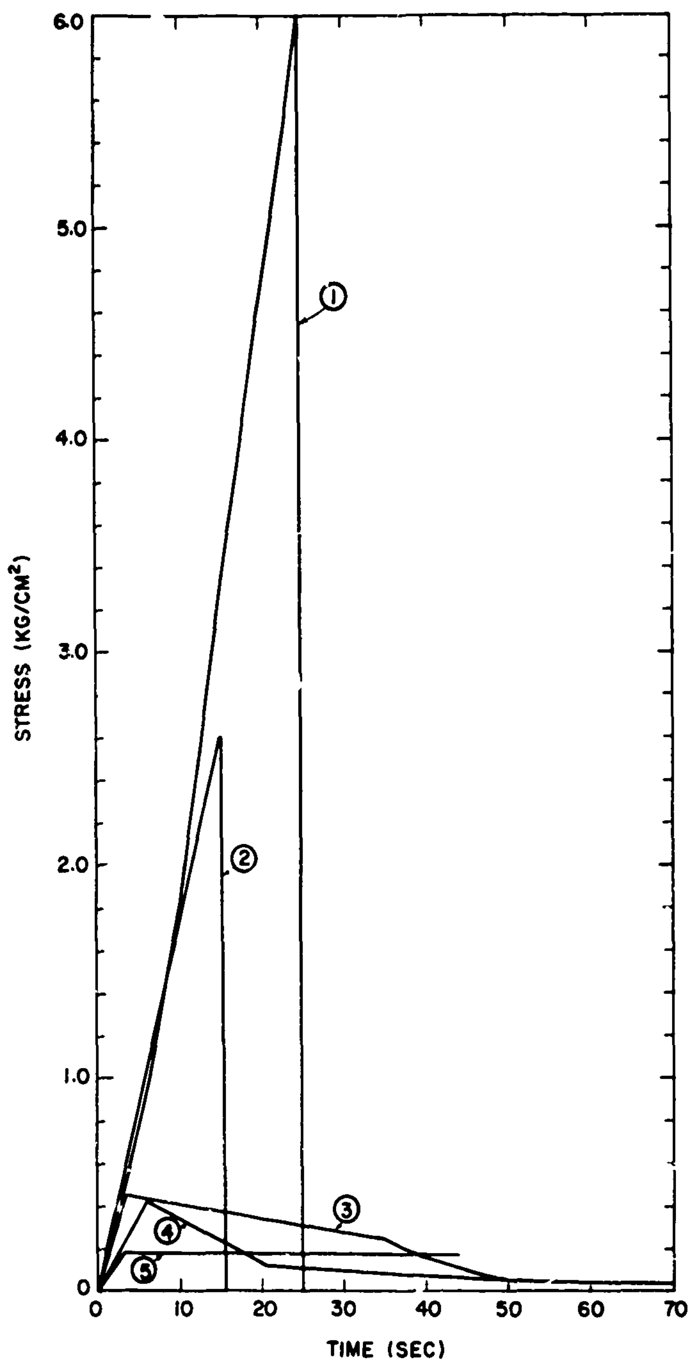

Figure 2. Typical stress vs time curves for stainless steel disks of different roughness and for a fused quartz optical flat. (Rates of shear are similar in all cases):

(1) Snow-ice/stainless steel, steel surface finished on lathe
(2) Snow-ice/stainless steel, mat polish
(3) Snow-ice/stainless steel, mirror polish
(4) Snow-ice/stainless steel, mirror polish
(5) Snow-ice/fused quartz optical flat (flat to within 1/5 waveband)
Temperature -4.5C.

Shear experiments were also performed to obtain adhesive strength as a function of the rate of shear (Table III). Average adhesive strength $\overline{S}$ (maximum values) as a function of the rate of shear $\underline{v}$ is shown in Figure 3.

The results can be expressed by,

$$\overline{S} = 69.9v + 0.22 \text{ kg/cm}^2 \qquad (1)$$

where $\underline{v}$ is expressed in cm/sec.

Table III. Adhesive strength of snow-ice sandwiched between stainless steel disks of mirror polish at -4.5C, as a function of the rate of shear. Cross-sectional area 6.16 cm^2. (For rate 5.8×10^{-3} cm/sec see Table 1c).

(a) Average rate 1.1×10^{-2} cm/sec.

Test no.	1	2	3	4	5	6	7	8	9	10	11	12
Adhesive strength (kg/cm^2)	0.51	0.82	0.55	1.09	0.92	1.06	1.23	1.13	0.94	0.57	0.70	0.75

Mean adhesive strength 0.86 kg/cm^2; standard deviation ± 0.23 kg/cm^2.

(b) Average rate 1.53×10^{-2} cm/sec.

Test no.	1	2	3	4	5	6	7	8	9	0	11	12
Adhesive strength (kg/cm^2)	0.95	0.72	0.84	1.29	0.99	1.86	1.32	1.53	1.87	2.04	1.26	1.45

Mean adhesive strength 1.34 kg/cm^2; standard deviation ± 0.42 kg/cm^2.

Ice/quartz interface

Experiments with ice frozen to a quartz surface were carried out at different rates of shear. As we have pointed out previously, the ice samples had a somewhat smaller cross section than the area of the fused quartz surface, which was flat within 1/5 of a lightband. The cross-sectional area of ice varied slightly according to the amount of ice melted when freezing it to the quartz surface: however, it remained practically circular and was measured at the end of each test.

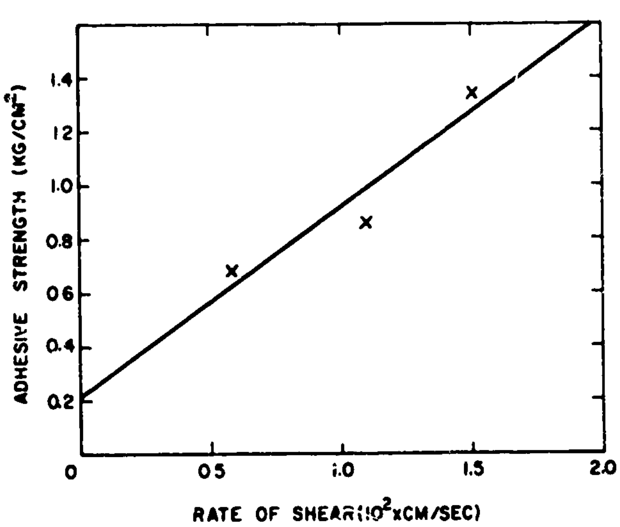

Figure 3. Relationship between average adhesive strength and rate of shear for snow-ice/stainless steel (mirror polish). Temperature -4.5C.

A typical stress vs time curve is shown in Figure 4, curve A. The stress rises quickly to a value which remains constant during the movement of the ice across the quartz surface. The distance travelled in about 20 seconds is about 1 to 2 mm. Even if the stress is taken off (Fig. 4, curve B) after some travel on the ice and reapplied after about 20 minutes, the new stress value is not changed appreciably. Table IV gives the experimental results for the adhesive strength as a function of shear rate.

Table IV. Adhesive strength of snow-ice (density 0.888 g/cm³) sandwiched between stainless steel and fused quartz (flat to 1/5 of a waveband) as a function of rate of shear at -4.5C. Cross-sectional area of quartz 5.07 cm². The cross-sectional area of ice differed slightly for each test but was smaller than the quartz area; it was measured at the end of each test. Height of snow-ice. 1 - 2 mm.

Test no.	Adhesive strength (kg/cm²)						
	Average rate of travel of quartz disk (cm/sec)						
	0.53×10^{-3}	1.1×10^{-3}	6.3×10^{-3}	1.2×10^{-2}	1.6×10^{-2}	2.5×10^{-2}	4.1×10^{-2}
1	0.063	0.058	0.147	0.151	0.440	0.225	1.06
2	0.066	0.204	0.262	0.325	0.220	0.264	0.84
3	0.098	0.132	0.279	0.239	0.238	0.441	1.04
4	0.073	0.099	0.174	0.154	0.359	0.471	0.70
5	0.053	0.058	0.129	0.106	0.422	0.344	0.74
6	0.087	0.081	0.330* 0.283†	0.172	0.585	0.203	0.63
7	0.099	0.248	0.144	0.111	0.275	0.427	0.50
8	0.047	0.090	0.176* 0.166†	0.228	0.435	0.558	0.59
9	0.060	0.135	0.171	0.205	0.322	0.614	0.73
10	0.091	0.096		0.248	0.310	0.477	0.48
11					0.286		0.56
Mean	0.074	0.120	0.184	0.194	0.355	0.402	0.72
Std. Dev.	±0.020	±0.059	±0.083	±0.063	±0.105	±0.130	±0.19

* First travel.
† Second travel after 20 min standing.

Figure 5 shows the average adhesive strength $\bar{S}$ (maximum values for higher rates of shear) plotted against the rate of shear v (evaluated by least squares). The straight line can be expressed by

$$\bar{S} = 15.1\,v + 0.07\ \text{kg/cm}^2 \qquad (2)$$

where v is in cm/sec. Figure 6 shows typical recordings at different rates of shear.

A number of tensile tests were carried out using the quartz plates and the tensile strength apparatus described in a previous report (Jellinek, 1957b). For this purpose the quartz optical flat was bonded by pliobond to a steel disk. The snow-ice cylinders of about 1 cm height and 2.3 cm diam were sandwiched between the quartz disk and a stainless steel disk. The breaks were cohesive and similar to those described in a previous report (Jellinek, 1957b). The results are given in Table V.

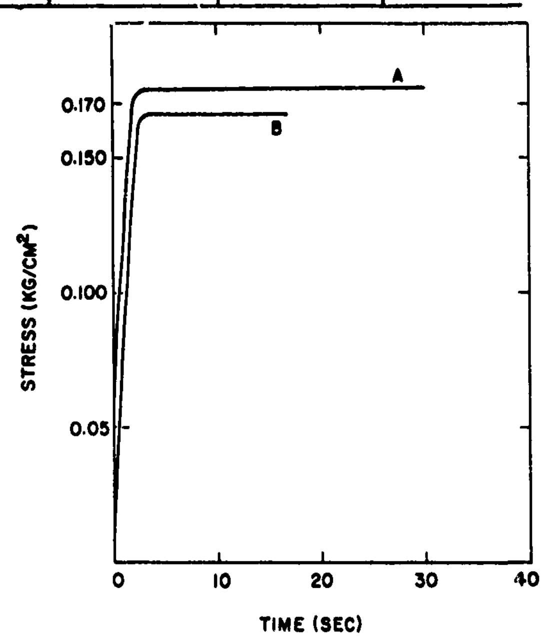

Figure 4. Adhesive strength vs time curve for snow-ice/fused quartz optical flat. A: First pull. B: Second pull after 20-min relaxation of stress. Temp -4.5C. Rate of shear 6.3×10^{-3} cm/sec. Cross-sectional area 3.36 cm².

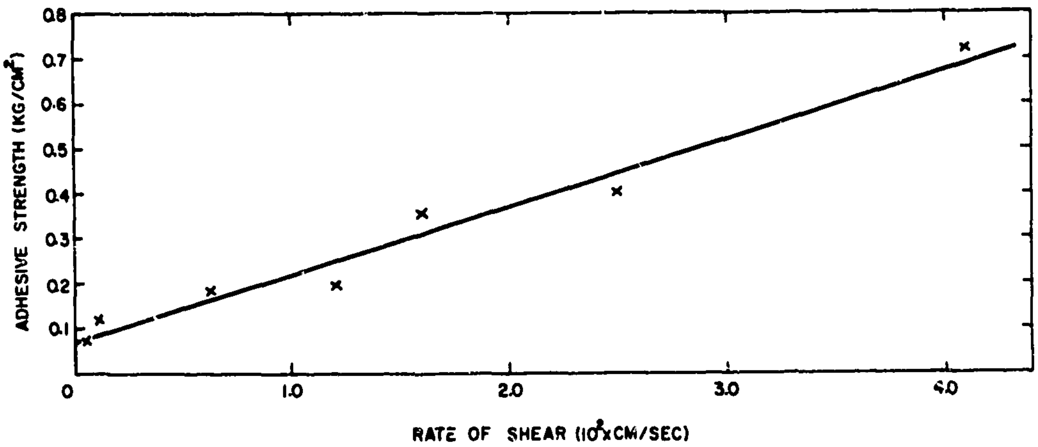

Figure 5. Relationship between average adhesive strength and rate of shear for snow-ice/fused quartz optical flat. Temp -4.5C.

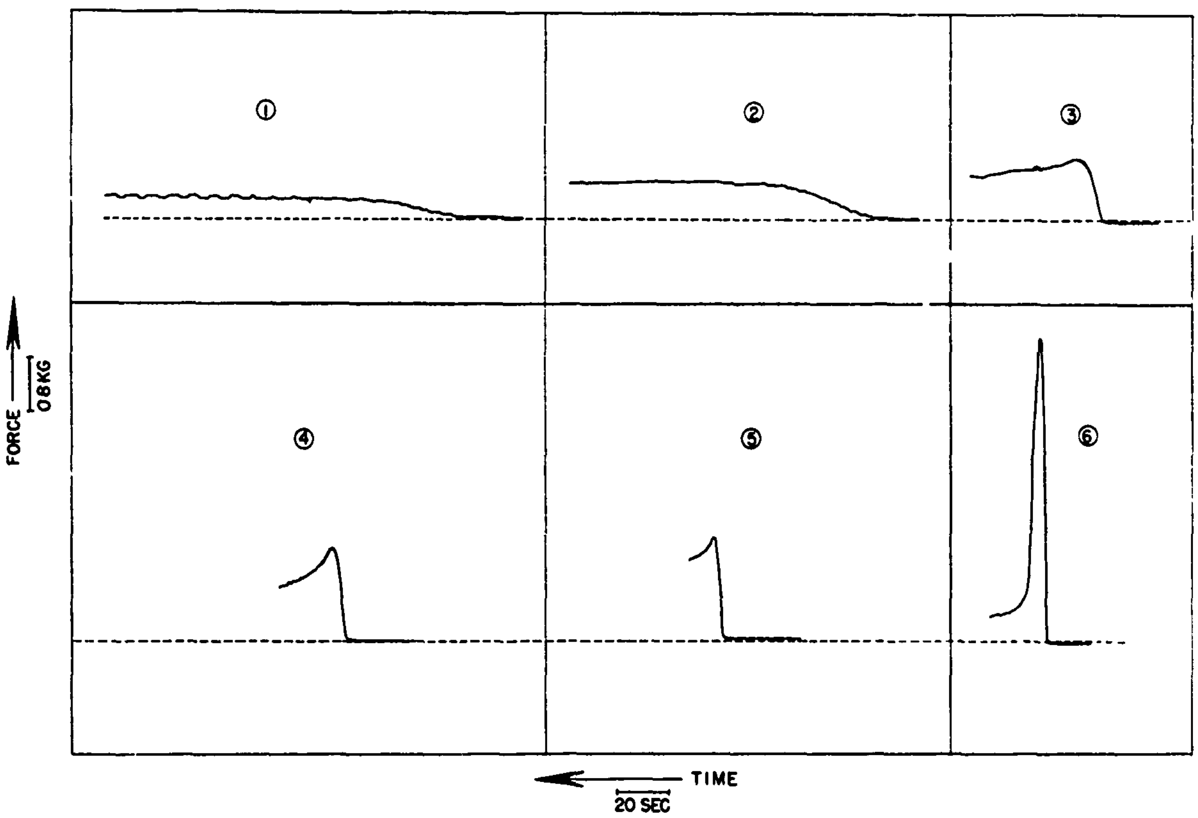

Figure 6. Typical recordings of force as a function of time at different rates of shear for snow-ice/fused quartz optical flat. Temp -4.5C.

(1) Rate 0.53×10^{-3} cm/sec; cross section 4.84 cm²
(2) " 1.1×10^{-3} cm/sec; cross section 4.24 cm²
(3) " 1.2×10^{-2} cm/sec; cross section 3.93 cm²
(4) " 1.6×10^{-2} cm/sec; cross section 4.84 cm²
(5) " 2.5×10^{-2} cm/sec; cross section 4.52 cm²
(6) " 4.1×10^{-2} cm/sec; cross section 4.84 cm²

Table V. Tensile strength experiments on the system snow-ice/fused quartz at -4.5C. Cylinder height about 1 cm. Diam about 2.3 cm. All breaks were cohesive.

Test no.	1	2	3	4	5	6
Tensile strength (kg/cm^2)	10.0	12.5	15.4	7.8	10.3	7.9

Mean tensile strength 10,6 kg/cm^2; standard deviation ±2.7 kg/cm^2.

DISCUSSION

The results presented in a previous report (Jellinek, 1957a) could be quite satisfactorily explained by the assumption of a liquidlike layer in the ice/metal interface. Thus on shearing, only frictional forces have to be overcome, whereas, in the case of tensile experiments, surface tension forces are involved, which are much larger than the frictional forces. In order to overcome the surface tension forces, a tensile stress has to be applied, which is given by the following equation:

$$\Delta p = \frac{2\gamma}{L} \tag{3}$$

where Δp is the pressure difference due to the curvature of the liquidlike film, γ the surface tension and $\underline{L}$ its thickness; L/2 is the radius of curvature (see Jellinek, 1957a) However, in the case of ice, Δp is larger than the tensile strength of ice, so that the break occurs in the ice (cohesive break) rather than in the liquidlike layer (adhesive break).

The results presented in this report substantiate the assumption of a liquidlike layer. The typical curves for ice/metal interfaces for stainless steel disks with surfaces of different degrees of roughness (Fig. 2) can be explained in the following way. The rougher the surface, the more ice wedges will be lodged in the metal surface; however, the liquidlike layer will still be present. On shearing, these ice wedges have to be broken, yielding a relatively high "adhesive" strength. With the more highly polished disks, the thickness of the liquidlike layer approaches that of the height of the unevenness in the metal surface; in addition there will be smaller and fewer ice wedges, hence the "adhesive" strength will decrease and gliding governed by frictional forces will eventually take place. The change of adhesive strength with rate of shear for mirror polished disks (Fig. 3) strongly suggests a process due to frictional forces in a liquid.

The experiments carried out with optically flat fused quartz plates show the whole phenomenon more clearly (Fig. 4, 6). At low rates of shear, the process shows the same characteristics as the shearing of a liquid between two flat plates. As the rate of shear increases, the shearing stress also increases. At high rates of shear, however, the character of the stress vs time curves changes (Fig. 6). The reason for this behavior is not quite clear, but it probably has something to do with the structure of the liquidlike layer. There will be an ordered transition region from the ice into the liquidlike layer, and one from the quartz into the liquidlike layer. Weyl (1951) has discussed the structure of such a layer. This structure has to be broken down on shearing and, at high rates of shear, the layer will be ruptured before it has time to adjust to the required movement. It is also probable that the liquidlike layer will contain some particulate matter (dust particles) which was present in the water and snow used for preparing the snow-ice.

The linear relationships between stress and rate of shear for the system ice/stainless steel and ice/quartz (Fig. 3, 5) are expressed by eq 1 and 2. The adhesive strength at zero rate of shear might possibly represent a yield value due to the structure of the liquidlike layer and the residual roughness of the stainless steel quartz respectively. The ratio of the viscosity to the thickness of the layer $\underline{L}$ can be calculated from the slopes of the straight lines considering the liquid to be Newtonian (this is a reasonable assumption except for the highest rates of shear).

Hence:

$$\frac{\eta}{L} = \frac{(\bar{S} - 0.22)}{v} = 69.9 \frac{\text{kg sec}}{\text{cm}} \quad \text{for ice/stainless steel}$$

$$\frac{\eta}{L} = \frac{(\bar{S} - 0.07)}{v} = 15.1 \frac{\text{kg sec}}{\text{cm}} \quad \text{for ice/quartz.}$$

In order to obtain the values for L and η separately, a second equation is needed. This is given in principle by eq 3, but, in the case of ice, Δp is larger than the tensile strength of ice and eq 3 cannot be utilized. However, it is reasonable to assume that the thickness of the liquidlike layer is approximately in the range of 10^{-5} to 10^{-6} cm at -4.5C. It would be unlikely to be 10^{-7} cm or even thinner. In that case even the optically flat quartz surface would be too rough. Similarly a thickness larger than 10^{-5}cm is not in accord with the results obtained on the mirror-polished stainless steel disks; profilometer readings showed the unevenness of these surfaces to be about 5 to 7μin. The viscosities obtained for the range of thickness from 10^{-5} to 10^{-6} cm are in the range of 70 to 700 poises for ice/stainless steel, and in the range of 15 to 150 poises for ice/quartz.

The tensile strength experiments employing the quartz plate are in agreement with the assumption of a liquidlike layer (Table V).

The conclusion reached on the basis of the experiments described in this report is that a liquidlike layer exists in the ice/stainless steel and ice/fused quartz interfaces. This liquidlike layer probably has a transition structure adjacent to the ice and a somewhat different one adjacent to the stainless steel or quartz.

It is well known that if a substance is subdivided, creating a large interfacial area, not only the bulk free energy has to be considered but also the interfacial free energy. Thus considering a small crystal sphere, the melting point of this sphere is lower than that of the solid in bulk because of the substantial contribution of the surface free energy. The relationship between the melting point and the radius of a small spherical crystal is given (Skapski, 1956) by

$$T_B - T_r = \frac{2T_B \gamma_{sl}}{q_f \rho_s r}$$

where T_B and T_r are bulk and small crystal freezing points respectively, on the absolute scale; r is the radius of the crystal (cm); q_f is the heat of fusion (ergs/g); ρ_s is the density of the crystal (g/cm^3), and γ_{sl} is the interfacial free energy of the crystal in contact with its liquid (ergs/cm^2). According to Skapski, the maximum decrease in melting point for ice is 43C for a radius $r_{min} = 1.12 \times 10^{-7}$ cm. The work of Sill (1954) is also of interest in this connection. His work is an elaboration of the work by Pawlov (1908), Tammann (1920), and Meissner (1920). Sill caried out experiments with stearic and myristic acids located in a thin wedge. Quite a good agreement was found between experiment and theory, showing that thin liquid layers can be in equilibrium with their solids at temperatures below their bulk freezing points because of their interfacial free energy. It is not unreasonable to expect that the surface free energy in the surfaces of solids in bulk will have an effect on the nature of their surface layer, especially for solids kept near their melting points. It is also of interest in this connection to mention Weyl's paper (1951) where he postulates an amorphous liquidlike layer on ice and discusses the structure of such a layer.

Recently experiments on adhesion of ice were carried out by Raraty and Tabor (1958). The experimental results of these authors substantially agree with the present ones. Torsional shear experiments were carried out with annular and cylindrical specimens. For annular specimens, the "adhesive strength" - temperature relation-

ship is similar to the one found by Jellinek (1957a). There is a linear increase of strength with decreasing temperature until a temperature is reached where the adhesive strength becomes larger than the cohesive strength; the latter is practically independent of temperature. For cylindrical specimens, Raraty and Tabor found that the linear increase in strength with decrease in temperature persists down to -25C, whereas Jellinek (1957a) found a transition point of adhesive to cohesive breaks at -13C. This discrepancy is probably due to the lower rates of shear used by Raraty and Tabor. Adhesion tests of ice were also carried out by these authors. For polystyrene for instance, they found an increase of adhesive strength with decreasing temperature; however, at about -15C the adhesive strength values became almost independent of temperature. Jellinek (1957a) found a linear increase of adhesive strength with decreasing temperatures, but his lowest temperature used was -15C. On the whole Raraty's and Tabor's results seem to fit into the picture of the process outlined above assuming a liquidlike layer.

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